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मानक

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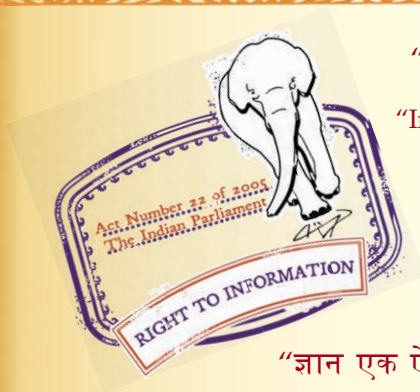
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“Step Out From the Old to the New”

IS 1448-96 (1980): Methods of Test for Petroleum and its Products, Part 96: Rust-preventing Characteristics of Steam-turbine Oil in the Presence of Water [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 1448 [P : 96] - 1980

Adopted : 19 March 1980

(Reaffirmed 2003)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 96]

RUST-PREVENTING CHARACTERISTICS OF STEAM-TURBINE OIL IN THE PRESENCE OF WATER

UDC 621·892·098 : 620·193

*Adapted from Joint Publication ASTM Designation D 665-60
(Reapproved 1977) and IP Designation 135/64 (Reapproved 1975)*

1. SCOPE

1.1 This method of test is intended to indicate the ability of turbine oils and heavier-than-water fluids, including those used for turbine gears, to aid in the prevention of rusting of ferrous parts, should water become mixed with the oil.

2. OUTLINE OF THE METHOD

2.1 The method involves stirring a mixture of 300 ml of the oil under test with 30 ml of distilled or artificial sea water, as required, at a temperature of 60°C with a cylindrical steel specimen completely immersed therein. It is customary to run the test for 24 hours; however, the test period may, at the discretion of the contracting parties, be for a shorter or longer period.

3. APPARATUS

3.1 **Oil Bath** — A thermostatically controlled liquid bath capable of maintaining the temperature of the oil sample at $60 \pm 1^\circ\text{C}$. An oil having a viscosity of approximately 35 cSt at 37·78°C is suitable for the bath. The bath shall have a cover with holes to accommodate the test beakers.

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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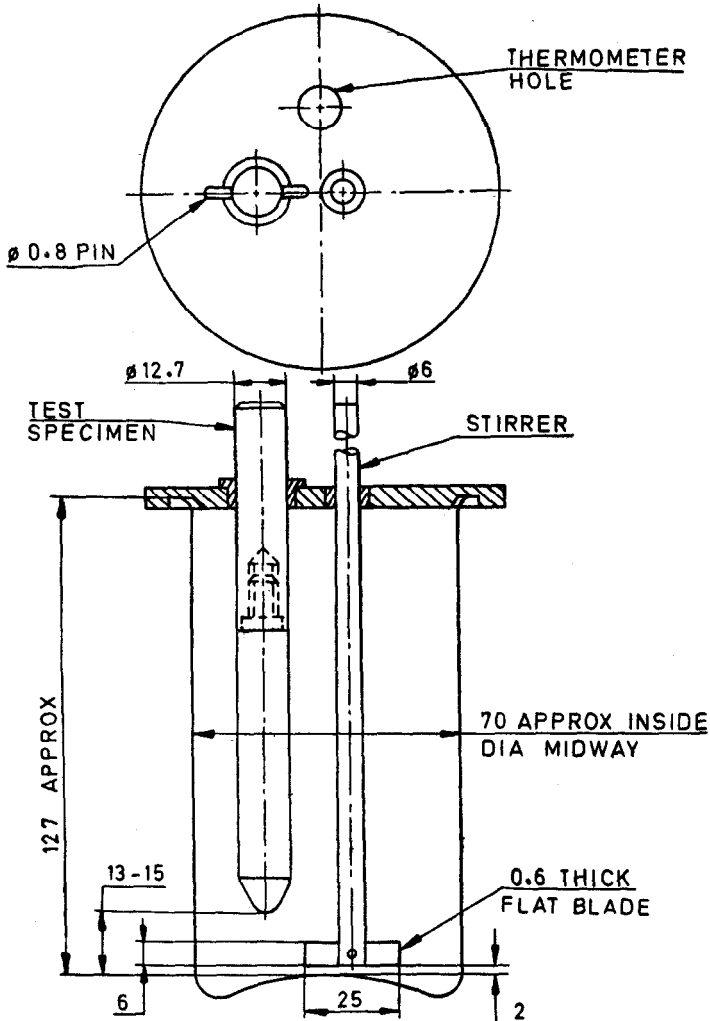
3.2 Thermometer — conforming to the requirements given below:

Range	25 to 105°C
Graduation	0.2°C
Immersion	50 mm
Overall length	410 $\pm$ 10 mm
Stem diameter	5.5 to 8.0 mm
Bulb shape	Cylindrical
Bulb length	10 to 20 mm
Bulb diameter	Not greater than stem
Length of graduated portion	240 to 280 mm
Distance, bottom of bulb to 25°C	95 to 110 mm
Longer lines at each	1°C
Figured at each	2°C
Expansion chamber	Required
Top finish	Ring
Scale error, <i>Max</i>	$\pm$ 0.2°C

3.3 Beaker — A 400-ml, tall-form heat-resistant glass beaker without pourout, as shown in Fig. 1, approximately 127 mm in height measured from the inside bottom centre and approximately 70 mm in inside diameter measured at the middle.

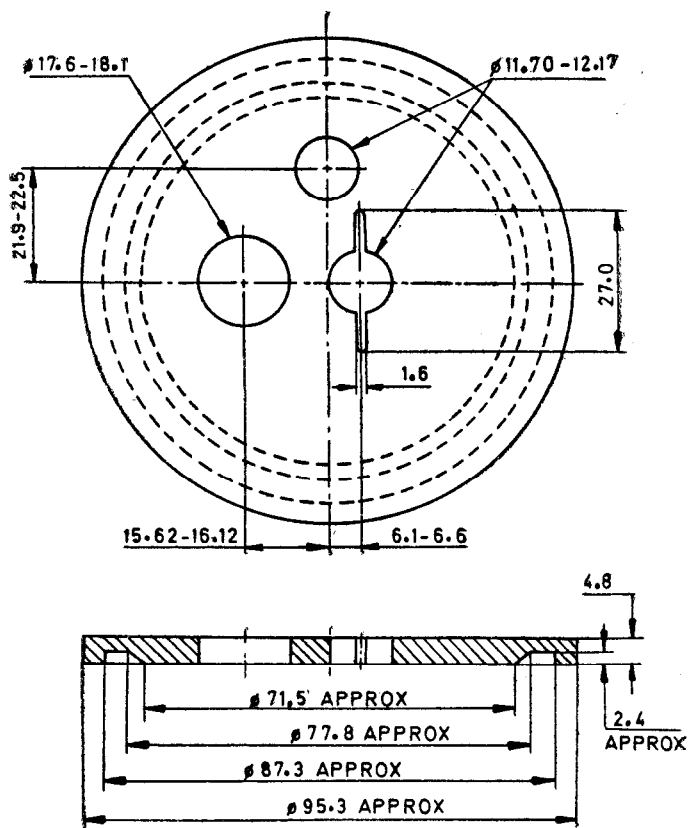
3.4 Beaker Cover — A flat beaker cover of glass or methyl methacrylate resin (*see* Note), kept in position, by suitable means such as a rim or groove. Two holes shall be provided on any diameter of the cover; one for a stirrer, 11.70 to 12.17 mm in diameter with its centre 6.1 to 6.6 mm from the centre of the cover; and the other, on the opposite side of the centre of the cover, for the test specimen assembly (*see* 4), 17.6 to 18.1 mm in diameter with its centre 15.62 to 16.12 mm from the centre of the cover. In addition, a third hole, 11.70 to 12.17 mm in diameter shall be provided for a thermometer, with its centre 21.9 to 22.5 mm from the centre of the cover and on a diameter of the cover at right angles to the diameter through the other two holes.

NOTE — An inverted Petri dish makes a suitable cover, as the sides of the dish aid in keeping it in position. Figure 2 shows a methyl methacrylate resin cover for the beaker which has been found to be suitable. An optional feature is shown, consisting of a slot, 1.6 mm by 27.0 mm which is centred on a diameter of the stirrer hole at right angles to the cover diameter through the specimen hole and stirrer hole. This feature allows withdrawal of the stirrer while the beaker cover is in place. When testing synthetic fluids, the beaker cover should be made from chemically resistant material such as chlorotrifluoroethylene.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR RUSTING TEST



All dimensions in millimetres.

FIG. 2 BEAKER COVER

3.5 Stirrer — A stirrer constructed entirely from stainless steel (see Notes 1 and 2) in the form of an inverted T. A flat blade $25 \times 6 \times 0.6$ mm shall be attached to a 6 mm rod in such a way that the blade is symmetrical with the rod and has its flat surface in the vertical plane.

NOTE 1 — The composition of stainless steel required shall be as follows:

<i>Constituent</i>	<i>Percent</i>	
Carbon (C), <i>Max</i>	0.16	
Silicon (Si), <i>Min</i>	0.20	
Manganese (Mn), <i>Max</i>	2.00	
Nickel (Ni)	7.0 to 10.0	} Both together 25.0, <i>Min</i>
Chromium (Cr)	17.0 to 20.0	
Sulphur (S), <i>Max</i>	0.045	
Phosphorus (P), <i>Max</i>	0.045	

NOTE 2 — If stainless steel is not available, stirrers made of heat-resistant glass and having approximately the same dimensions as the stainless steel stirrers specified may be used.

3.6 Stirring Apparatus — Any convenient form of stirring apparatus capable of maintaining a speed of $1\,000 \pm 50$ rev/min.

3.7 Grinding and Polishing Equipment — A 150 and 240 grit metal working aluminium oxide abrasive cloth (or equivalent) closed coat on a jeans backing, suitable chuck (*see* Fig. 3) for holding the specimen; and a means of rotating the specimen at a speed of 1 700 to 1 800 rev/min.

4. TEST SPECIMEN AND ITS PREPARATION

4.1 The specimen assembly shall consist of a round steel specimen fitted to a plastic holder. The plastic holder shall be made of methyl methacrylate resin in accordance with the dimensions shown in Fig. 4 (two types of holders are illustrated). When testing synthetic fluids, the plastic holder should be made from chemically resistant material such as tetrafluoroethylene. The round steel specimen when new shall be 12.7 mm in diameter and approximately 68 mm in length exclusive of the threaded portion which screws into the plastic holder and shall be tapered at one end as shown in Fig. 4. It shall be made of steel conforming to the following chemical composition:

<i>Constituent</i>	<i>Percent, Max</i>
Carbon (C)	0.25
Silica (Si)	0.35
Manganese (Mn)	1.00
Sulphur (S)	0.060
Phosphorus (P)	0.060

4.1.1 If these steels are not available, other equivalent steels may be used, provided they are found to be satisfactory by comparative test. The steel specimen, either new or from a previous test (*see* Note), shall be prepared as described in 4.2 and 4.3.

NOTE — When making a check test, the steel specimen that shows rust should not be re-used. Specimens that repeatedly show rust in tests of various oils may be imperfect. Such specimens should be used with oils known to pass the test. If rusting occurs in repeat tests, these specimens should be discarded.

4.2 Preliminary Grinding — If the specimen has been used previously and is free from rust or other irregularities, preliminary grinding may be omitted, and it may be subjected only to final polishing as prescribed in 4.3. If the specimen is new or if any part of its surface shows rust or other irregularities, clean it with petroleum hydrocarbon solvent (60/80) and grind with medium 150 grit aluminium oxide cloth to remove all irregularities, pits, and scratches, as determined by visual inspection (*see Note*). Perform the grinding by mounting the specimen in the chuck of the grinding and polishing apparatus, and turning it at a speed of 1 700 to 1 800 rev/min while the 150 grit aluminium oxide cloth is applied. Old 150 grit aluminium oxide cloth may be used to remove rust or major irregularities, but the grinding shall be completed with a new cloth. Proceed at once with the final polishing with 240 grit aluminium oxide cloth, or remove the specimen from the chuck and store in *iso*-octane until needed. Discard re-used specimens when the diameter is reduced to 9.5 mm.

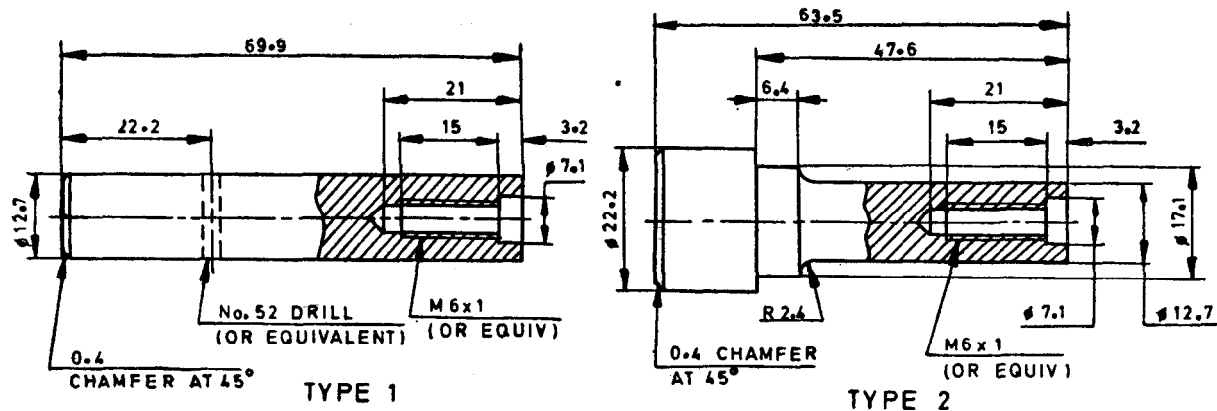
NOTE — The specimen shall not be touched with hands at any stage after cleaning with the petroleum hydrocarbon solvent (which precedes either preliminary grinding or final polishing) until the test is completed. Forceps or a clean, lintless cloth may be used for handling.

4.3 Final Polishing — Just before the test is to be made, subject the specimen to final polishing with 240 grit aluminium oxide cloth. If the preliminary grinding has just been completed, stop the motor which rotates the specimen. Otherwise, remove the specimen from the *iso*-octane (previously used unruined specimens shall be stored, in this reagent), dry with a clean cloth and place in the chuck. Rub a new piece of 240 grit aluminium oxide cloth longitudinally over the static specimen until the rounded end, and the entire surface show visible scratches. Rotate the specimen at a speed of 1 700 to 1 800 rev/min and polish with a strip of 240 grit aluminium oxide cloth by wrapping it halfway around the specimen, and applying a firm but gentle downward pull to the loose ends of the cloth for about 1 to 2 minutes, so as to produce a uniform finely scratched surface free from longitudinal scratches. Carry out the final stage of the polishing with new cloth (*see Note*). Remove the specimen from the chuck without touching with fingers, wipe lightly with a clean, dry, lintless cloth or tissue (or brush the specimen lightly with a camel hair brush), attach to the plastic holder, and immediately immerse in the oil to be tested. This may be either the hot oil sample (*see 5.1*) or a clean test tube containing a portion of the sample. The specimen may be removed later from this tube and allowed to drain briefly before being placed in the hot oil.

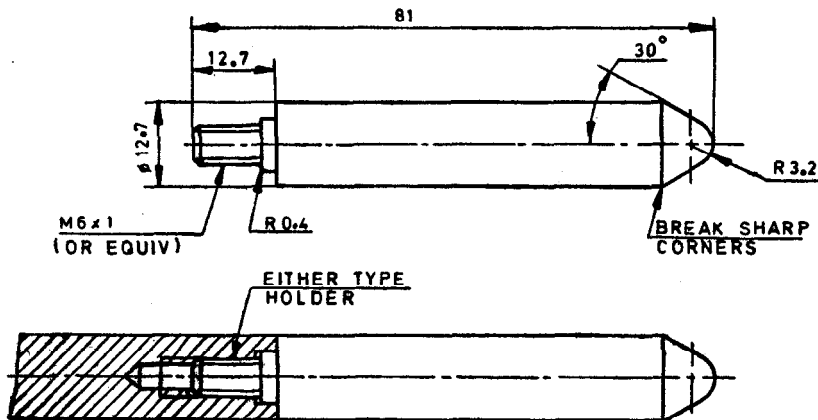
NOTE — To ensure that the flat shoulder (that portion of the specimen perpendicular to the threaded stem) is free of rust, this area should be polished. This may be done by holding a strip of 240 grit of aluminium oxide cloth between the chuck and the shoulder while rotating the specimen for a brief period.



FIG. 3 CHUCK FOR POLISHING TEST SPECIMENS



SPECIMEN HOLDERS



All dimensions in millimetres.

FIG. 4 TEST SPECIMEN ASSEMBLY

5. PROCEDURE A (FOR DISTILLED WATER)

5.1 Clean the beaker with chromic acid cleaning solution, wash with distilled water to remove the acid completely, and dry in an oven. Clean a glass beaker cover and a glass stirrer by the same procedure. To clean a stainless steel stirrer and a methyl methacrylate resin cover, use petroleum hydrocarbon solvent (60/80) wash thoroughly with hot water and finally with distilled water, and dry in an oven at a temperature not over 65°C for the cover. Pour 300 ml of the oil to be tested into the beaker and place the beaker in the oil bath held at a temperature that will maintain $60 \pm 1^{\circ}\text{C}$ in the oil sample. The beaker shall be inserted into a hole of the bath cover and suspended in the hole with the beaker rim resting on the bath cover. The oil level in the bath shall not be below the oil level in the test beaker. Cover the beaker with the beaker cover with the stirrer in position in the proper opening. Adjust the stirrer so that the shaft is 6 mm off centre in the beaker containing the oil sample and the blade is within 2 mm of the bottom of the beaker. Then suspend a thermometer through the hole in the cover intended for that purpose so that it is immersed to a depth of 57 mm. Start the stirrer and when the thermometer reading reaches $60 \pm 1^{\circ}\text{C}$ insert the steel specimen prepared in accordance with 4.

5.2 Insert the test specimen assembly through the specimen hole in the beaker cover and suspend so that its lower end is 13 to 15 mm from the bottom of the beaker. Either type plastic specimen holder (Fig. 4) may be used. The hole through which the specimen is suspended shall be unobstructed (*see* Note).

NOTE — Figure 1 shows the arrangement of the apparatus.

5.3 Continue stirring for 30 minutes to ensure complete wetting of the steel specimen. With the stirrer in motion, remove the thermometer temporarily and add 30 ml of distilled water through this hole, discharging the water on the bottom of the beaker, and replace the thermometer. Continue stirring at a speed of $1\,000 \pm 50$ rev/min for 24 hours (*see* Note) from the time water was added, maintaining the temperature of the oil-water mixture at $60 \pm 1^{\circ}\text{C}$. Stop stirring at the end of 24-hour period, remove the specimen, allow to drain, and then wash with the petroleum hydrocarbon solvent. If desired, the specimen may be preserved by lacquering.

NOTE — In general, observations of rusting made at the end of 12 hour testing time are indicative as to whether the oil will pass or fail. It is customary to run the test for 24 hours, however, the test period may, at the discretion of the contracting parties, be for a shorter or longer period.

6. PROCEDURE B (FOR ARTIFICIAL SEA WATER)

6.1 The procedure for rust-preventing characteristics of steam turbine oils in the presence of artificial sea water shall be identical with that described in 5.1, 5.2 and 5.3, except that artificial sea water shall be used

in place of distilled water in that portion of the procedure described in 5.3. The artificial sea water shall have the following composition:

<i>Salt</i>	<i>g/l</i>
NaCl	24.54
MgCl ₂ .6H ₂ O	11.10
Na ₂ SO ₄	4.09
CaCl ₂	1.16
KCl	0.69
NaHCO ₃	0.20
KBr	0.10
H ₃ BO ₃	0.03
SrCl ₂ .6H ₂ O	0.04
NaF	0.003

6.2 The solution may be conveniently prepared as described below. The method avoids any precipitation in concentrated solutions with subsequent uncertainty of complete resolution. Using chemicals and distilled water, prepare the following stock solutions:

<i>Stock Solution No. 1:</i>	<i>g</i>
MgCl ₂ .6H ₂ O	3 885
CaCl ₂ (anhydrous)	406
SrCl ₂ .6H ₂ O	14

Dissolve and dilute to 7 litres.

<i>Stock Solution No. 2:</i>	
KCl	483
NaHCO ₃	140
KBr	70
H ₃ BO ₃	21
NaF	2.1

Dissolve and dilute to 7 litres.

6.2.1 To prepare the artificial sea water, dissolve 245.4 g of sodium chloride (NaCl) and 40.94 g of sodium sulphate (Na₂SO₄) in a few litres of distilled water, and add 200 ml of stock solution No. 1 and 100 ml of stock solution No. 2 and dilute to 10 litres. Stir the 10-litre batch and add 0.1 N sodium carbonate (Na₂CO₃) until the pH is between 7.8 and 8.2. One or two millilitres of the carbonate solution will be required.

7. REPORT

7.1 Since the test may be performed either by Procedure A or Procedure B and since it may be conducted for various periods of time, reports of results should indicate the procedure followed, for example, Rusting Test, 24 hours with distilled water; or, Rusting Test, 48 hours with artificial sea water.

7.2 All inspections at the end of the test to determine the condition of specimens shall be performed without magnification under normal light. For the purpose of this test, normal light is considered to be illumination of about 645·8 lux. Within the meaning of this method, a rusted specimen is one on which any rust spot or streak is visible by the above inspection procedure.

7.3 In order to report an oil as passing or failing, the test shall be conducted in duplicate. An oil shall be reported as passing the test if both specimens are rust-free at the end of the test period. An oil shall be reported as failing the test if both specimens are rusted at the end of the test period (*see Note*). If one specimen is rusted while the other is free of rust, tests on two additional specimens shall be made (*see Note under 4.1.1*). If either of these latter specimens shows rusting, the oil shall be reported as not passing the test. If neither of these latter specimens shows rusting, the oil shall be reported as passing the test.

NOTE — An indication of the degree of rusting occurring in this test may be desired. For uniformity in such cases, use of the following classifications of rusting severity is recommended:

Light rusting — Rusting confined to not more than six spots, each of which is 1 mm or less in diameter.

Moderate rusting — Rusting in excess of the above but confined to less than 5 percent of the surface of the specimen.

Severe rusting — Rusting covering more than 5 percent of the surface of the specimen.

8. PROCEDURE FOR HEAVIER-THAN-WATER FLUIDS

8.0 General — The stirring action provided by the stirrer prescribed in 3.5 is not sufficient to thoroughly mix the water and test sample when the fluid under test is heavier-than-water. The following clauses define changes in the method for testing heavier-than-water fluids. Except as specifically stated, all the requirements of the previous clauses 1 to 7 shall apply.

8.1 Apparatus

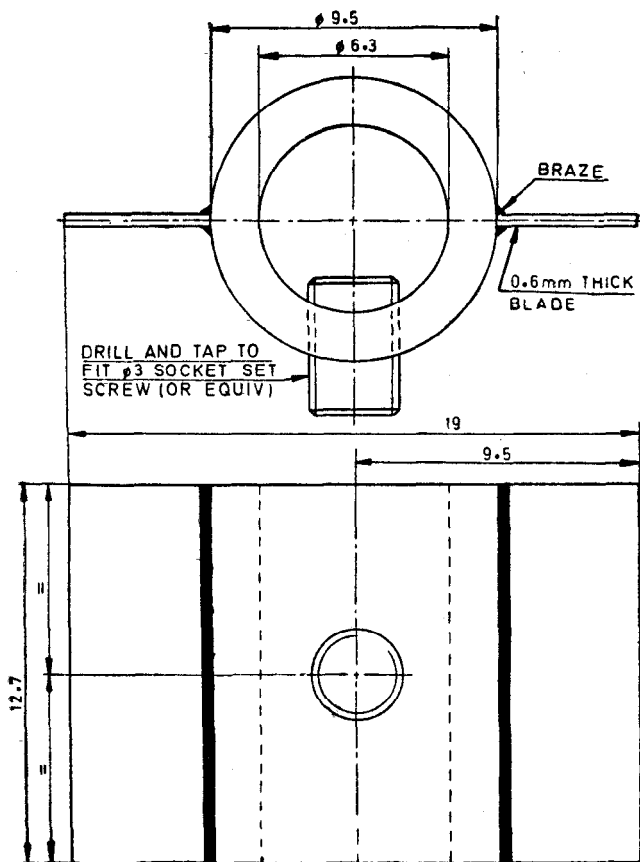
8.1.1 Beaker Cover — Same as prescribed in 3.4 (*see Note*).

NOTE — Some heavier-than-water fluids may attack or dissolve methyl methacrylate beaker covers and test specimen holders. It is recommended that chlorotri-fluoroethylene beaker covers and tetrafluoroethylene specimen holders be used when testing heavier-than-water fluids.

8.1.2 Stirrer — Same as prescribed in 3.5 but with an auxiliary blade attached to the stirrer shaft. The auxiliary blade shall be of stainless steel, $19.0 \times 12.7 \times 0.6$ mm, as shown in Fig. 5. The auxiliary blade shall be positioned on the stirrer shaft so that the bottom edge of the auxiliary blade is 57 mm above the top edge of the fixed lower blade, and so that the flat surfaces of both blades are in the same vertical plane.

8.2 Test Specimen and Its Preparation

8.2.1 Same as prescribed in 4.1 (see Note under 8.1.1).



All dimensions in millimetres.

FIG. 5 AUXILIARY BLADE FOR STIRRER